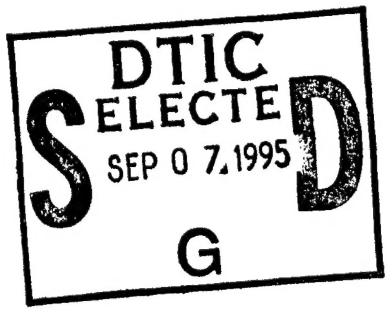
Heteroepitaxial Diamond Growth

Final Report
1 January 1994- 31 December 1994



Submitted to
Ballistic Missile Defense Organization
Innovative Science and Technology Office
Office of Naval Research
Program No. N00014-92-C-0081

Prepared by
Research Triangle Institute

83U-5294
September 1995

REPORT DOCUMENT PAGE

Form Approved
OMB No 0704-0188

1. AGENCY USE ONLY *(Leave blank)*	2. REPORT DATE	3. REPORT TYPE AND DATES COVERED
	September 1995	Final Report 1 January 1994 -- 31 December 1994

4. TITLE AND SUBTITLE

Heteroepitaxial Diamond Growth

5. FUNDING NUMBERS

N00014-92-C-0081

6. AUTHOR(S)

R. J. Markunas, R. A. Rudder, J. B. Posthill, R. E. Thomas, G. Hudson

7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES)

Research Triangle Institute
P. O. Box 12194
Research Triangle Park, NC 27709

8. PERFORMING ORGANIZATION REPORT NUMBER

83U-5294

9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESSES(ES)

Office of Naval Research
800 N. Quincy Street
Arlington, VA 22217-5000

10. SPONSORING/MONITORING AGENCY REPORT NUMBER

11. SUPPLEMENTARY NOTES

12a. DISTRIBUTION/AVAILABILITY STATEMENT

Approved for public release; unlimited distribution

12b. DISTRIBUTION CODE

13. ABSTRACT

This phase of the program saw progress in four key technology areas: (1) homoepitaxial diamond optimization, (2) epitaxial joining to form a tiled array, (3) diamond single-crystal lift-off, and (4) negative electron affinity (NEA) of diamond surfaces. These results are summarized.

14. SUBJECT TERMS

15. NUMBER OF PAGES

16. PRICE CODE

17. SECURITY CLASSIFICATION OF REPORT	18. SECURITY CLASSIFICATION OF THIS PAGE	19. SECURITY CLASSIFICATION OF ABSTRACT	20. LIMITATION OF ABSTRACT
UNCLASSIFIED	UNCLASSIFIED	UNCLASSIFIED	

TABLE OF CONTENTS

Final Report

1 January 1994- 31 December 1994

1.0 INTRODUCTION

This is the 1994 Final Report on the Heteroepitaxial Diamond Growth Program Contract No. N-00041-92-C-0081.

This phase was exceptionally productive in advancing the component technologies that are required for production of a single-crystal diamond boule and diamond wafers. The technical details for each process area have already been discovered in the respective 1994 quarterly reports, and we briefly list and summarize the program highlights herein. An abstract (accepted for presentation) is included that will provide a means to disseminate our results publicly.

2.0 SUMMARY OF ACCOMPLISHMENTS IN THIS PHASE

The major achievement in this phase break out in four areas. Three of these areas are necessary components for a diamond single-crystal boule and wafer technology to be realized. Negative electron affinity (NEA) studies of diamond surfaces is the other major area of achievement. The reader is referred to the listed 1994 quarterly reports for complete technical details.

2.1 Homoepitaxial Diamond

RTI is now able to grow state-of-the-art (100) homoepitaxial diamond using water/alcohol mixtures. The growth rate is ~ 0.5 μm/hr., the surfaces are topographically smooth as observed by FEG-SEM, Raman shows no 1332 cm^{-1} line broadening when compared with native diamond single crystals and the etch-pit density is in the mid -$10^5 cm^{-2}$ to low-$10^6 cm^{-2}$ range. More technical details can be found in First, Second and Third 1994 Quarterly Reports.

2.2 Epitaxial Joining to Form a Tiled Array

Developing a diamond epi-compatible process to bond crystallographically-oriented diamond crystals in close proximity (micron separation) was the key to a successful demonstration of epitaxial joining. Two 3 mm × 3 mm (100) face and edge oriented crystals were joined epitaxially using our homoepitaxial diamond process. We foresee no intrinsic obstacles to scaling this to larger areas and more crystals in order to create a diamond single-crystal template. The defect density is higher in the joint region, but further development that incorporates higher crystallographic tolerances may lead to reduced defect densities. More technical details can be found in Second and Third 1994 Quarterly Reports.

2.3 Diamond Single-crystal Lift-Off

One large-area single-crystal diamond template would be little value if it could not be thickened and wafers cut from it. In other words, a critical part of any single-crystal boule technology is the ability to cut wafers from it. While diamond can be cut and/or cleaned, it can be desirable to do this with minimal loss of materials (kerf loss) and on planes other than the natural cleavage plane of diamond, (111). We have combined our diamond (100) homoepitaxial process with a technology developed at NRL (M. Marchywka, et al.) to demonstrate such a so-called "lift-off" process of cutting single-

crystal C(100) wafers. More technical details on this process can be found in the Fourth 1994 Quarterly Report.

2.4 Negative Electron Affinity (NEA) of Diamond Surfaces

NEA of H-terminated diamond was "discovered" fortuitously in our laboratory while investigating the electrical properties of epitaxial Ni contacts on type IIb (naturally B doped) diamond. This has led to a series of SEM and surface spectroscopy experiments and manuscripts being produced. These results indicate that diamond has great potential to be a component part of a cold electron emitter. The technical details of this work are included in all four of the 1994 Quarterly Reports.

2.5 Conclusion

This phase has demonstrated experimentally the success of several component technologies that lead to a diamond single-crystal and wafer fabrication process. Additionally, NEA has been observed on suitably prepared diamond surface and explained scientifically. Both of these areas have potential technological utility -- depending on future DoD system requirements.

3.0 PUBLICATION

1. J. B. Posthill, D. P. Malta, T. P. Humphreys, G. C. Hudson, R. E. Thomas, R. A. Rudder, and R. J. Markunas, *Development of Epitaxial, Tiling, and Cutting Processes for a Diamond Single-crystal Wafer Technology*, accepted for presentation, 1995 Fall Materials Research Society Conference, Boston, MA.

DEVELOPMENT OF EPITAXIAL, TILING, AND CUTTING PROCESSES FOR A DIAMOND SINGLE-CRYSTAL WAFER TECHNOLOGY J.B. Posthill, D.P. Malta, T.P. Humphreys, G.C. Hudson, R.E. Thomas, R.A. Rudder, and R.J. Markunas, Research Triangle Institute, Research Triangle Park, North Carolina 27709-2194.

Development of a diamond homoepitaxial deposition process that utilizes water and ethanol at a growth temperature of ~600°C is described. Topographies are excellent, and etch pit densities (EPD) are in the 10^6 cm^{-2} range when growth is done on type Ia C(100) substrates. This process has been used to epitaxially join diamond single-crystals that were bonded in close proximity to each other. This process of "tiling" single-crystal diamonds in close proximity in order to manufacture a large-area diamond single-crystal template is also described. Specially prepared diamonds that have had their faces and edges oriented to {100} were used. Heteroepitaxial Ni-coated diamond surfaces are pressed onto a Si wafer while their edges are pressed together while being heated in an inert gas atmosphere. The resulting bond is excellent; permitting our 600°C diamond deposition process to epitaxially join the diamonds. A diamond wafer technology also requires the ability to cut with minimal kerf loss and cost, which has been addressed using a specific sequence consisting of: ion implantation, homoepitaxial diamond growth, annealing, and contactless electrochemical etching. This "lift-off" method of cutting has thus far resulted in a 2 mm×0.5 mm×17.5μm transparent, synthetic, free-standing, single-crystal diamond plate being fabricated. Raman and EPD show the plate to be comparable to our best homoepitaxial diamond.

Accepted for presentation, 1995 Fall Materials Research Society Conference, Boston, MA.